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16. ABSTRACT

This paper presents the preliminary results of compatibility testing of various grouts with selected hazardous wastes. The compatibility testing is a part of an ongoing project to determine the geotechnical feasibility of utilizing selected grouts and state-of-the-art techniques in the bottom sealing of existing hazardous waste sites. All of the grout set times appear to be affected to some degree by being mixed with the selected waste solutions on a 1 to 1 volume basis. The urethane grout tested was the most sensitive to the presence of the various waste solutions.

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GEOTECHNICAL ASPECTS OF BOTTOM SEALING EXISTING HAZARDOUS WASTE LANDFILLS BY INJECTION GROUTING

(Phase I - Compatibility Studies)

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INTRODUCTION

Background. Eighty percent of the EPA listed priority hazardous waste dumps (total 418) threaten contamination of city water supplies with toxic waste (Dombrowski, 1982). Numerous other waste sites across the country are leaching contaminants into the ground water. Because of the high cost of totally cleaning up existing waste sites, the in situ containment of hazardous waste is being considered as a cost-effective remedial action. Bottom sealing by injection grouting is one of the methods being evaluated. In situ containment of hazardous waste by injection of chemical or cementiceous grouts has been presented in various reports (Kitchens, 1980; Spooner, et. al. 1983) as a means of eliminating or minimizing pollution from waste disposal sites. Most of the discussions of containment methods are general in nature because of the variability of hazardous materials and types of pumpable grouts amenable to a variety of geological conditions. If an ideal combination of the geologic, chemical waste and grout type variables were to occur at a particular waste disposal site, adequate stabilization could be accomplished. The technical feasibility of stabilizing the bottom of a variety of waste disposal sites, using a variety of grouts, can only be determined by controlled laboratory and pilot studies. The Waterways Experiment Station (WES) has technical expertise in the areas of hydrogeology, hazardous waste site investigations, and grouting which is needed to adequately perform the necessary bench and pilot studies. Grouting of waste disposal sites after emplacement of some or all wastes is a recent concept in effectual control of hazardous waste materials, primarily liquids. Theoretically grouts can be used to permeate voids and gel (set) to effectively reduce the permeability of the geological material and/or waste at a site. The grouting techniques that can be employed are void sealing, area blanket grouting, jet pancake grouting, curtain grouting, and contact grouting (Bowen, 1981). The various grouting techniques are briefly described below:

Large void sealing. Grouts have been used extensively in the past for sealing large voids in rocks and other materials associated with dams and tunnels. Void sealing grouting methods could be adapted to seal between a slurry wall and highly fractured or weathered bedrock at waste disposal sites or to seal between certain types of waste and the bottom of the disposal pits.

Area blanket grouting. Area grouting is accomplished by injecting grout into a series of closely spaced injection holes placed in a grid pattern. The closely spaced holes enable the individual grout pods to coalesce and form an impermeable blanket beneath a waste site (Figure 1, Tolman, et. al. 1978). The grout has to be of sufficient viscosity to penetrate and fill the voids in the underlying geologic material or under enough pressure to hydrofrac and form a seal.

Jet pancake grouting. Jet grouting is a method of emplacing grout into "pancake like" voids cut into soil or soft rock by water or grout emitted from a high-pressure nozzle. Using this technique it is theoretically possible to emplace a continuous grout seal beneath a waste site (Huck, et. al. 1980).

Curtain grouting. A grout curtain or wall can be formed around a waste site by drilling a pattern of borings in triple rows of primary and secondary holes. Starting with the primary holes grout is injected to form pods or pillows, in stages working upward or downward. The grouting of the primary hole forms columns of grout which are then tied together by injecting columns of grout into the secondary holes. The coalescing columns form an impermeable wall or curtain (Hayward-Blake, et. al. 1980).

Contact grouting. Low injection pressure contact grouting has been used in the past to seal the sides of excavations and to patch cracks in concrete dams or dikes. Contact grouting could possibly be effective for encapsulation of the contents of a small site.

The objective(s) of virtually every grouting project is to increase the strength of a permeable geologic material and/or control the flow of fluids through a material. The diversion or cessation of fluid flow through a permeable material via grouting has many applications in construction, ground-water control and liquid waste migration control. In most instances the flow is through earth material having a specific permeability subject to fluid migration in response to gravity. The effect of the response is that the fluid flows down the hydrologic gradient. Similarly, liquid hazardous waste leaking from ruptured drums or tanks in a disposal site is subject to gravitational flow which results in liquids flowing through the bottom of the disposal area. Vertical flow downward through the disposal area base is most prevalent in areas where the bottom of the disposal site is above the ground water (water table) surface. The effectiveness of a grout to prevent the migration of a chemical liquid waste through a permeable material depends upon the grout-chemical waste compatibility, grout emplacement feasibility, and grout-earth material compatibility. A recent study by JRB Associates concerning available information on the compatibility of grouts with hazardous wastes indicated that very little actual chemical compatibility testing has been performed (JRB Associates, 1982).

Purpose and Objective. The purpose of the WES study is to determine the geotechnical feasibility of utilizing selected grouts and state-of-the-art techniques in solving the problems associated with the in situ bottom sealing of existing hazardous waste sites. The geotechnical feasibility of bottom sealing by grouting is mainly dependent on two variables: (1) the physical criteria for emplacing the grout at the desired location, and (2) the compatibility of grouts and hazardous wastes. The overall WES effort involves a graduated study beginning with small-scale laboratory tests and concluding in a large-scale pilot grouting operation in a controlled environment.

The objective of the sealing or stabilization of the base of a hazardous waste landfill is to minimize or eliminate the migration of contaminants from the disposal area. The stabilized zone may include the bottom of the waste, the underlying geologic media and/or contaminated ground water.

This paper deals primarily with the compatibilities of various grouts with selected hazardous waste.

Approach. To underseal a hazardous waste landfill, it is necessary to develop data concerning the chemical interaction between grout and the waste materials and between grout and the contaminated ground water beneath a landfill. This phase of the WES investigation includes the following:

a. Determining normal grout gelling (setting) time.

b. Determining changes in gelling time produced when grouts were mixed with laboratory prepared solutions representing contaminated ground water (10% or saturations of selected chemical).

c. Measuring gelled (or set) grout deterioration as indicated by decreased strength and/or increased permeability when gellation occurs in contaminated ground water.

d. Measuring grout deterioration when exposed to chemically contaminated ground water for an extended period of time (20 days).

PROPERTIES OF GROUTS

Cement. Cement grouts are suitable for rock and structures grouting, but are limited in soil (less than 0.1 mm dia. voids) grouting because the large particle size of the cement mixture reduces the permeability of the soil. Although special agents may be added to prevent excessive water bleeding and alter the rate of setting, the problem of particle size still limits the use of cements. Water-cement ratio, degree of setting, and additives all affect the physical properties of cured and fresh cement (concrete). The properties of strength and durability are a function of time duration, temperature, humidity and compatibility with the material grouted (earth materials). Properties of the cement grout can be deliberately altered by mixing with other grout types such as acrylamide. The chemical compatibility data of cement grouts with various chemical compounds is not available in literature, however, the compatibility data of cement components with selected compounds is available. In general, cement grouts are compatible with hydroxides, weak alkali solutions, heavy metals and neutral salts (JRB, 1982).

Silicate Grouts. Silicate grouts, the second most commonly used grout, typically employ an alkali silicate, a gelling agent, and water. Gelling agents consists of acids, polyvalent cations, acid-forming compounds or selected organic compounds (Kirk-Othmer, 1979, Hurley and Thornburn, 1971). To enhance gelling, accelerators such as chlorides, aluminates or bicarbonates may be used (Johnson, 1979). Organic esters may be used as a catalyst for retarding gelling time (Bowen, 1981). The viscosities of

sodium silicate grouts vary from 1.5 to 50 cP and may be as high as 260 cP (Sommerer and Kitchens, 1980, and Tollard and Caron, 1977). Silicate grouts are subject to deterioration through synersis, dessication and solution erosion by ground water (Hurley and Thornburn, 1971). These properties and the varying grout mixture ratios can result in physical degradation such as fissure formation. Physical degradation will then increase the surface area available to chemical degradation. Silicate grouts once gelled are resistant to moderate amounts of acids and alkali solutions (Kirk-Othner, 1979).

Organic Polymer Grouts. The two types of grouts selected to represent this grout group are acrylamide and urethane grouts. The polymerization and crosslinking of these organic monomers forms an insoluble gel which is resistant to many waste chemicals. Acrylamide grouts consist of a monomer base, a crosslinking agent, an initiator and an accelerator. Linear polymers result from reactions at the double bond site in the acrylamide and crosslinking of these linear structures are accomplished at the amide group. The degree of this crosslinking determines the grout properties (Tollard and Caron, 1977a). With viscosities as low as 2 cP, the acrylamide grouts are capable of being injected into fine sediments.

SELECTION OF MATERIALS AND METHODS

Selection of Materials. To determine the compatibility of commercially available grout types with the thousands of available chemicals and mixtures of chemicals expected in a hypothetical disposal site would be prohibitive due to time and cost factors. However, basic grout types can be tested with selected chemicals representing the generic chemical types expected to be found in the waste of larger industries. This information, heretofore unavailable, would provide the foundation for possible grout applications in liquid hazardous waste control. The selection of four representative grouts resulted from a thorough literature review. Available literature emphasized the advantages of all types of commercially available grouts including those fulfilling the market objectives of grouts no longer available due to toxicity problems. Relative toxicity of grouts (Table 1) along with commercial availability and chemical inertness were the primary considerations in selecting the four grout types. Portland cement was selected because of its continuing to be the most common grout material employed despite its limited application in finer grained sediment. The acrylamide grout represents those grouts replacing the AM-9 grout which dominated the chemical grouting market in the early 70's. The silicate grouts have the longest history of usage which extends back to 1886. The urethane grout represents the second largest chemical grout commonly in usage.

The secondary considerations of grout types selected included the set times and viscosity (Table 2). The substrate of landfills and spill areas are naturally variable requiring that grouts of varying viscosities be tested. Chemical grouts being much less viscous than cement grouts can be injected at least 10 times as fast (Bowen, 1981). As a representative material to be grouted, sand was selected. The sand performed as a container of the grout and permitted chemical/grout compatibility testing. The sand selected was concrete sand from a local distributor. Concrete sand being of mixed size fractions will not replicate any specific landfill substrate, but was selected to represent a general earth material media. Many existing landfills are emplaced in alluvial sand and gravel. Landfill substrate could vary considerably from the selected test sand in mineral composition and permeability, however, these variables are not expected to affect the grout-waste compatibility.

Representative chemicals were selected from the large spectrum of available chemicals representing waste stream outputs of major industries. The representative chemicals, nature and industry are listed in Table 3. These organic and inorganic compounds, if improperly disposed of, have sufficiently low viscosity to infiltrate soil materials and enter the ground water. Once in the ground water, even those wastes considered insoluble, could have soluble constituents or additives. The factors of migration and groundwater contamination are different for every disposal site depending on such factors as soil absorption, ground-water flow, etc. In this study ground-water dilution was assumed to be 10 to 1 by weight, water to waste chemical.

Methods. To determine the effect of water/chemical solutions on grout gell time, it was necessary to determine grout gell times according to manufacturer's recommended or CE-specified mixture procedures. This gell time determination was conducted on .25 liter samples. The samples after being thoroughly mixed were allowed to gel on the gelometer apparatus. The geolometer blade rotates in the grout mixture until the preset shear stress is reached in the grout solution. When the shear stress is reached the gelometer timer is automatically stopped. Gelation time is then recorded by the lab technician. The gelometer was not used to determine set time for the cement grout mixtures. Cement grouts were

tested on the Ferioli (Malano, Italy) which indicates initial and final set time.

Emplacement of the respective grouts in field situations may partially or totally be in zones not contaminated by waste solutions. This may result from inadvertently or intentionally injecting grout in a zone not yet contaminated. These grouted zones may later be subjected to migrating contaminants. To determine the resistance of chemical and cement grouts to chemical wastes after the grouts reach final set, laboratory tests were performed on cylindrical grout slugs (3.2 cm by 2.5 cm dia) or near cubic specimens (3.2 cm X 3.2 cm) (Figure 2). Each grout type sample was cured in a mold of polyvinylchloride (PVC). For each grout type, a slug of grout and grout with sand was cast in a PVC mold prior to immersion in the chemical waste solutions. The results of the slugs reaction to chemical wastes were interpreted via physical changes in the specimens. The results of the interaction were generally of four categories; expansion, dissolution, shrinkage or no observable change (Table 4).

To obtain workable grout gelling times for laboratory tests, manufacturer specified alterations of mixtures were followed. The manufacturers procedures were also followed in larger tests wherein extended times were required to permit time for mixing and injection. Mixing in the larger scale tests was done inline in the grouting equipment. The grout gel and cement set times are the baseline times for comparison with grouts and cement added to the contaminated aqueous solutions. Solutions of the selected wastes were prepared prior to grout mixture preparation. The chemical (Table 5) aqueous solutions were of 10% concentration or saturation, whichever occurred first. The solutions were then added to the grout mixture in 1:1 ratio and gellation or set times were determined. The 1:1 ratio was selected because grouting beneath a hazardous waste site in a saturated zone of waste contaminated ground water may approach this worst condition mixing with grout. Figure 3 shows examples of four grout types mixed with twelve chemical waste solutions. The response of bentonite grout to the various waste solutions is also being addressed (Figure 4). The large-scale tests included gr * injection into and above waste saturated sand. The grouts selected for injection were those performing best in the laboratory grout waste compatibility testing. Ten to twenty liters of each grout was injected into drums of sand (Figure 5) saturated with waste solutions and control drums of sand saturated with potable water. The grout pods (inside the drums) were developed during grouting by injecting grout and withdrawing the grout injection tube simultaneously. The controlled grout injection and withdrawal of the grouting tube produced pods of grouted sand. The grout was not permitted to contact the inner drum wall. Contact of grout with the drum wall would have possibly affected grout flow paths. Such an impedence to grout flow would not have simulated grout injection in a natural unconfined subsite earth material. The grouted pods were removed from respective drums and sampled. Sampling consisted of cutting test specimens for strength, compatibility and permeability testing. Determinations of permeability and physical strength will be performed by the Geotechnical and Structures Laboratories, respectively.

SUMMARY OF LABORATORY STUDIES

The set times for the four selected grouts varied from grout type to grout type as well as within the same grout type with variations in the amounts of grout components added. The acrylamide grout set is approximately 30 seconds, cement initial sets varied from 5 to 7 hours, silicate grout sets varied from 25 to 192 minutes, and the urethane grout set time was from 3 to 19 minutes. A range of set times was sought to facilitate planning of drum and large-scale grout injection tests and as background data for mixing grouts with the chemical waste solutions.

Table 4 represents data on the physical changes in grout samples placed in a chemical waste bath after the grouts had completely set. Examination and recording of physical changes in the grout samples was performed after 20 days.

Tables 6-10 provide data on the effects of waste solutions on grout set times. Set times of the grout material alone are provided along with the set times after the grout was mixed with waste solutions on a 1:1 ratio by volume. The mixing preceded the initiation of setting whenever possible. In some instances setting was possibly initiated during mixing without being observed.

PRELIMINARY CONCLUSIONS

Compatibility data is critical in determining the feasibility of using injection grouting to bottom seal an existing hazardous waste site. All the grout set times appear to be affected to some degree by being mixed with the selected waste solutions on a 1:1 volume basis. The urethane grout tested was the most sensitive to the presence of the various waste solutions. The previously set grout samples which were immersed in chemical waste/water solutions responded in many instances by shrinking, swelling, or dissolving. All the silicate grouts tested set to some degree when injected into waste solution

saturated sand. The average pod diameter of silicate grout injected into concrete grade sand was 40.85 centimeters for an 8.5 liter injection of grout followed by a 5.6 liter injection placed 15 centimeters above the first.

The phased approach of the investigation, i.e., going from small-scale laboratory tests to gradually larger scale pilot tests, has been effective in producing the desired results while reducing the number of larger more expensive tests needed for final evaluation.

FUTURE WORK

Based on the results of the injection of grout into saturated sand and laboratory bench scale testing of grout-grout bonding characteristics, a larger scale test is planned. This larger scale test will include the injection of grout pods into large sand-filled metal containers to produce a bowl-shaped concave layer. The grouting will be conducted in several stages to test the effectiveness of the grout in producing an effective impermeable barrier to water. The water will be added uniformly over the grouted section. Water migrating through the grout layers will be collected and volumes measured.

Subsequently an even larger pilot test will be conducted to determine the largest possible grout pod which can be developed for each respective grout type. Four such pods will be produced adjacent to one another. Excavation of the pods will be conducted to determine size and bonding characteristics.

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Table 1

Toxicity of Selected Grouts Oral LD_{50} (mg/Kg) for Rats

Grout Type	Components	Components	Set Grout
Silicate	-	-	>15,000
	Sodium Silicate	1100	-
	Calcium Chloride	1000	-
	Magnesium Chloride	2800	-
	Dimethylformamide	1500	-
Acrylamide	-	-	5,000
	Acrylamide Monomer	200	-
	Mechylenebiracrylamide	390	-
Urethane	-	-	5,000
	Toluene Diisocyanate	5800	-
	Acetone	9750	-
Cement	Portland Cement	Nontoxic	Nontoxic

Sources: Tallard and Caron, 1977b
Berry, 1982
Geochemical Corporation, 1982

Table 2

Ranges of Values of Various Properties of Selected Grouts

Grout Type	Viscosity (cp)	Setting Time (min)	Strength (gm/cm^2)
Silicate	1.5 - 50	0.1 - 3000	1 - 150
Acrylamide	1.2 - 1.6	0.1 - 1000	20 - 200
Urethane	20 - 200	0.08 - 120	NA

NA: Not Available

Sources: Sommerer and Kitchens, 1980
Tallard and Caron, 1977a
Tallard and Caron, 1977b
Karol, 1982a
Bowen, 1981
Avanti International, 1982

Table 3

Hazardous Wastes

Waste Type	Nature	Product
Hydrochloric Acid	Acid	Descaling Steel Pickle Liquor
Sodium Hydroxide	Base	Metal Cleaning
Ammonium Hydroxide	Base	Chemical Industry
Ammonium Chloride	Salt	Chemical Industry
Copper Sulfate	Salt	Plating Waste
Potassium Chromate	Salt	Metal Cleaning
Benzene	Cyclic Hydrocarbon	Degreaser
Trichloroethylene	Halogenated Hydrocarbon	Degreaser
Phenol	Substituted Benzene	Paint Stripper
Toluene		Paint Thinner
Gasoline	Hydrocarbon Mixture	Fuel
Oil	Hydrocarbon Mixture	Lubricant

Table 4

Grout Slug/Waste Compatibility After 20 Days

Waste	Grout Types			
	Acrylamide	Cement	Silicate	Urethane
Potassium Chromate	SW	NC	SH	SH
Hydrochloric Acid	SH	NC	NC	SH
Ammonium Hydroxide	SW	NC	SH	SW
Sodium Hydroxide	SW	NC	D	D
Toluene	SW	NC	SH	SW
Cupric Sulfate	SH	NC	NC	SH
Ammonium Chloride	SW	NC	SH	SH
Benzene	SW	NC	NC	SW
Trichloroethylene	SW	NC	SH	SW
Phenol	SW	NC	NC	NC
Gas (Unleaded)	SW	NC	SH	SW
Oil (SAE 30)	SW	NC	SH	SW

NC = No Change

SH = Shrink

SW = Swell

D = Dissolve

Table 5

Waste Solutions

10% Solutions	Saturated (<10% Solution)*
Ammonium Chloride	Benzene
Ammonium Hydroxide	Oil (SAE 30)
Cupric Sulfate	Phenol
Hydrochloric Acid	Toluene
Potassium Chromate	Trichloroethylene
Sodium Hydroxide	Unleaded Gasoline

* Saturated solutions resulted from low solubilities of waste chemical added.

Table 6

Acrylamide Grout/Waste Set Data

	Waste Solution	Percentage	Set Time
A.	Potassium Chromate	10%	No Set
B.	Hydrochloric Acid	10%	No Set
C.	Ammonium Hydroxide	10%	No Set
D.	Sodium Hydroxide	10%	No Set
E.	Toluene	Saturated	Partial Set/ 1 hr. 25 min.
F.	Cupric Sulfate	10%	No Set
G.	Ammonium Chloride	10%	5 Minutes
H.	Benzene	Saturated	5 Minutes
I.	Trichloroethylene	Saturated	7 Minutes
J.	Phenol	Saturated	No Set
K.	Gas (Unleaded)	Saturated	7 Minutes
L.	Oil (SAE 30)	Saturated	7 Minutes

Acrylamide grout set time approximately 30 seconds.

Table 7

Silicate/Waste Mixture Set Data - 30% Grout Solution

	Waste Solution	Percentage	Set Time
A.	Potassium Chromate	10%	42 minutes
B.	Hydrochloric Acid	10%	4 minutes
C.	Ammonium Hydroxide	10%	2 hrs., 30 min.
D.	Sodium Hydroxide	10%	Does Not Set
E.	Toluene	Saturated	1 hr., 30 min.
F.	Cupric Sulfate	10%	Set on Contact
G.	Ammonium Chloride	10%	Set on Contact
H.	Benzene	Saturated	2 hrs., 12 min.
I.	Trichloroethylene	Saturated	1 hr., 54 min.
J.	Phenol	Saturated	3 minutes
K.	Gas (Unleaded)	Saturated	1 hr., 10 min.
L.	Oil (SAE 30)	Saturated	1 hr., 3 min.

Silicate grout set time 41.9 minutes.

Table 8

Silicate/Waste Mixture Set Data - 50% Grout Solutions

	Waste Solution	Percentage	Set Time
A.	Potassium Chromate	10%	23 minutes
B.	Hydrochloric Acid	10%	Set On Contact
C.	Ammonium Hydroxide	10%	3 hours
D.	Sodium Hydroxide	10%	3 hrs., 20 min.
E.	Toluene	Saturated	2 hrs., 15 min.
F.	Cupric Sulfate	10%	Flocked On Contact
G.	Ammonium Chloride	10%	Flocked On Contact
H.	Benzene	Saturated	2 hrs., 30 min.
I.	Trichloroethylene	Saturated	3 hours
J.	Phenol	Saturated	Hardened On Contact
K.	Gas (Unleaded)	Saturated	3 hours
L.	Oil (SAE 30)	Saturated	5 hrs., 30 min.

Silicate grout set time 25.2 minutes.

Table 9

Urethane Grout/Waste Set Data

	Waste Solution	Percentage	Set Time
A.	Potassium Chromate	10%	No Set
B.	Hydrochloric Acid	10%	"
C.	Ammonium Hydroxide	10%	"
D.	Sodium Hydroxide	10%	"
E.	Toluene	Saturated	"
F.	Cupric Sulfate	10%	"
G.	Ammonium Chloride	10%	"
H.	Benzene	Saturated	"
I.	Trichloroethylene	Saturated	"
J.	Phenol	Saturated	"
K.	Gas (Unleaded)	Saturated	"
L.	Oil (SAE 30)	Saturated	"

Urethane grout set time 3.3 minutes.

Table 10

Portland Cement/Waste Mix

	Waste	Percentage	IST*	FST+
A.	Potassium Chromate	10%	4	7.5
B.	Hydrochloric Acid	10%	5	~24
C.	Ammonium Hydroxide	10%	5	9.5
D.	Sodium Hydroxide	10%	3	7.5
E.	Toluene	Saturated	5	7.5
F.	Cupric Sulfate	10%	IST when mixed	~24
G.	Ammonium Chloride	10%	--	~24
H.	Benzene	Saturated	4.5	7.5
I.	Trichloroethylene	Saturated	3	7.5
J.	Phenol	Saturated	45.	7.5
K.	Gas (Unleaded)	Saturated	4	8
L.	Oil (SAE 30)	Saturated	4.5	8

Recipe was for a .7 Portland mix, initial set - 6 hrs and final set - 11.5 hrs.

Tested hardness of samples with hand-held penetrometer.

IST* = Initial Set Time in Hours
FST+ = Final Set Time in Hours

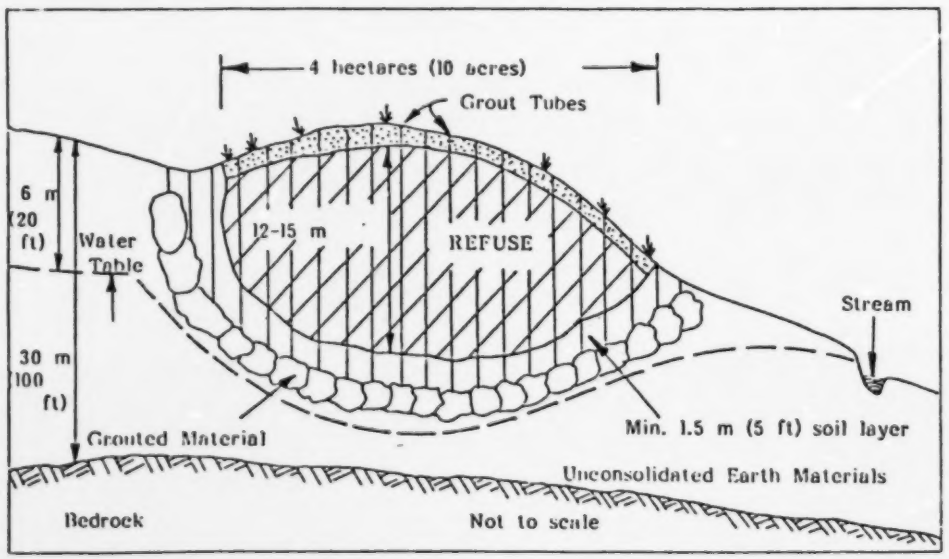

Figure 1. Cross sectional of grouted bottom seal beneath a landfill (Tolman, et al., 1978)

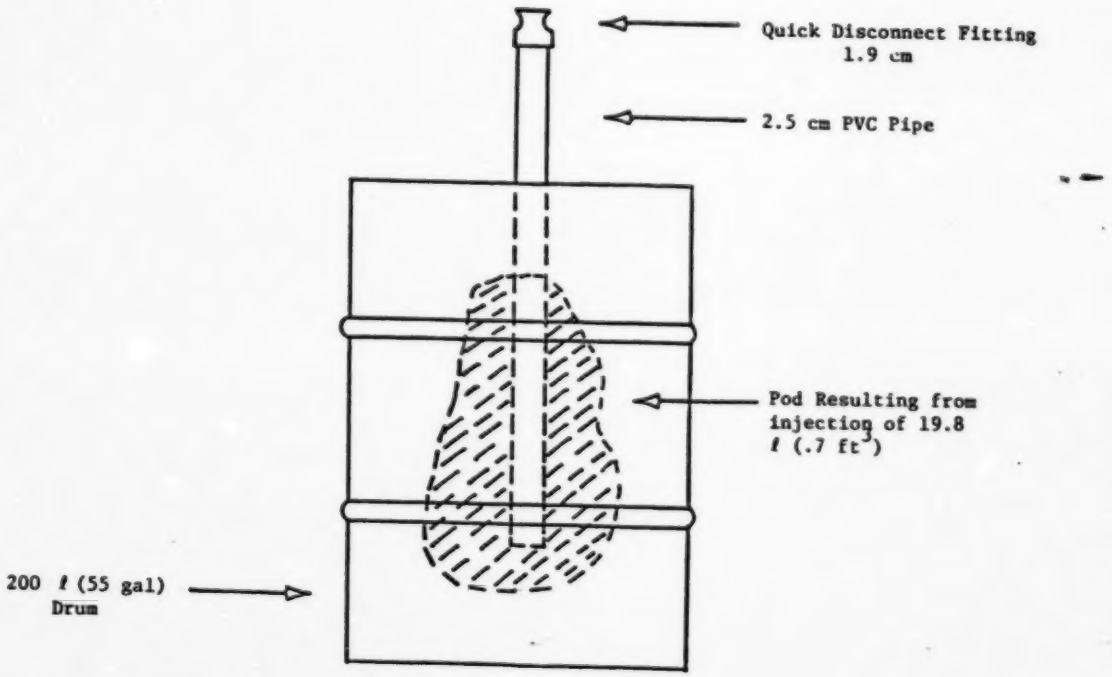

Figure 5. Grout injection small-scale test

Initiation of grouting was at the base of the PVC pipe, one foot from the bottom of the drum. Each successive injection was 6" higher than the previous one

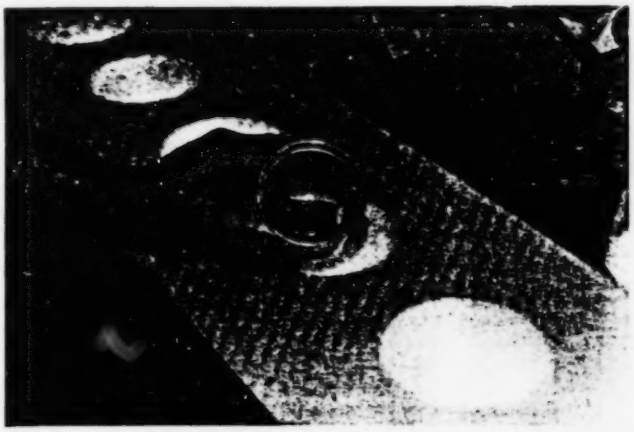

Figure 2. Slug of grout in chemical waste/water solution after approximately 60 days. No significant change in shape had occurred with this sample.

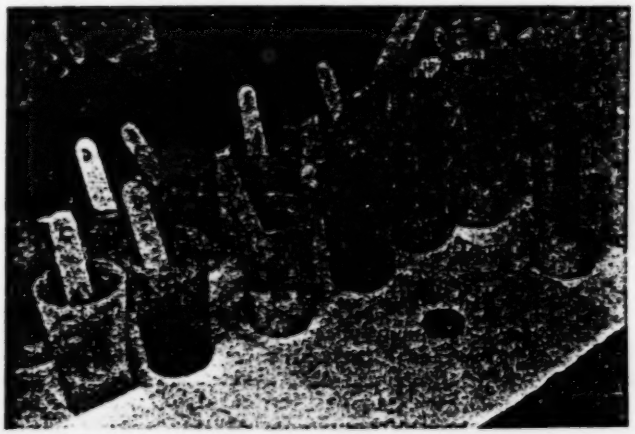

Figure 3. Samples of four grout types mixed with the 12 chemical waste/water solutions on a 1:1 ratio.

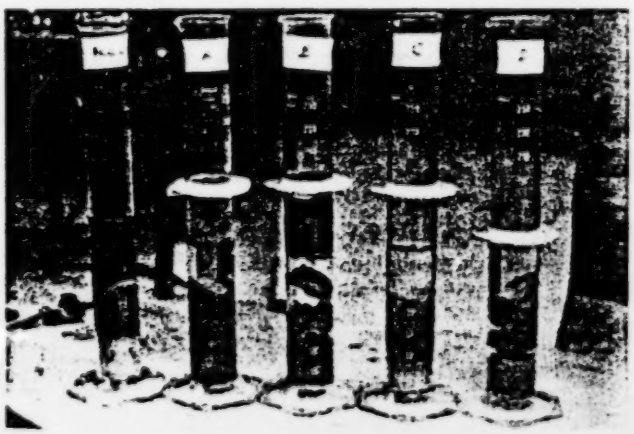

Figure 4.

Response of bentonite to waste solutions. Blank sample is bentonite with deionized water; Sample A is bentonite with potassium chromate, B is with hydrochloric acid, C is with ammonium hydroxide and D is with sodium hydroxide.

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